



UNIVERSITI PUTRA MALAYSIA

**FLAVOUR CHARACTERIZATION OF JACKFRUIT (*ARTOCARPUS
HETEROPHYLLUS* L.) FROM FIVE CULTIVARS AND
OPTIMIZATION OF CANNING CONDITIONS FOR
JACKFRUIT PUREE**

ONG BEE TEIN

FSTM 2006 11

**FLAVOUR CHARACTERIZATION OF JACKFRUIT (*ARTOCARPUS
HETEROPHYLLUS* L.) FROM FIVE CULTIVARS AND OPTIMIZATION OF
CANNING CONDITIONS FOR JACKFRUIT PUREE**

By

ONG BEE TEIN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of Requirement for the Degree of Master of Science**

May 2006



DEDICATION

*To my dearest mother, Yeong Siew Yong,
for being so patient and understanding,
My father, Ong Sin Ken,
for providing me with trust and liberty,
My brothers, Lieh Bin, Lieh Chee and Lieh Yan,
for their strong support and brotherly care,
My sisters-in-law,
for their guidance and advice,
And last but not least my boyfriend,
for being there always in my times of need,
tolerating me with his patience*

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

FLAVOUR CHARACTERIZATION OF JACKFRUIT (*ARTOCARPUS HETEROPHYLLUS* L.) FROM FIVE CULTIVARS AND OPTIMIZATION OF CANNING CONDITIONS FOR JACKFRUIT PUREE

By

ONG BEE TEIN

May 2006

Chairman: Nazimah Sheikh Abdul Hamid, PhD

Faculty: Food Science and Technology

The study concerns flavour characterization of five jackfruit (*Artocarpus heterophyllus* L.) cultivars and optimization of canned jackfruit puree production. In the first part of this study, twenty three volatile compounds extracted using dichloromethane solvent extraction were tentatively identified by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). As ripening progressed, there was an increase in volatile compound formation. Development of new volatile flavour compounds in trace amounts at day 3 after harvest indicated the start of jackfruit ripening. Data obtained showed that the ripening process of jackfruit was at its optimum at day 5 after harvest. Variation of volatile compounds in different portions (top, middle and bottom) of the fruit during ripening was too little to give any significance.

The volatile profiles of jackfruit flavour in five cultivars were established using headspace solid phase microextraction (SPME) and gas chromatography-time of flight mass spectrometry (GC-TOFMS). Qualitative and quantitative analyses were carried out using divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) fiber with an extraction time of 10 min. Thirty seven compounds were identified from the five cultivars tested. Characteristic aroma which are in higher concentrations and contributed to jackfruit flavour were found to be ethyl isovalerate, 3-methylbutyl acetate, 1-butanol, propyl isovalerate, isobutyl isovalerate, 2-methylbutanol, and butyl isovalerate. The consistent occurrence of these compounds in all cultivars of jackfruit suggested their importance in contributing to the sweet and fruity note of jackfruit. Concentration of the volatile compounds present played an important role in determining the overall flavour of each fruit cultivar. Each cultivar also possessed its own unique compound which distinguished them from one to another.

During the canning of jackfruit puree, optimization of time (10 – 30 min) and temperature (100 – 120°C) was studied using response surface methodology. Response values fitted the second order polynomial model. Effect of the independent variables on sweetness, pH and total soluble solids were insignificant ($p > 0.05$). Analysis of variance (ANOVA) and regression coefficients showed significant positive linear effect of temperature towards cooked flavour ($p < 0.01$), sourness ($p < 0.05$), bitterness ($p < 0.05$) and hue ($p < 0.05$), while negative linear effect was seen in fruity flavour ($p < 0.05$), viscosity ($p < 0.01$) and chroma ($p < 0.001$). Influence of processing time on quality characteristics of jackfruit puree was only found in cooked flavour ($p < 0.05$) and hue

($p < 0.05$). The optimization model generated a desirable region which depicted optimal processing temperature to be in the range of 100 °C and 110 °C while processing time to be in the range of 15 min to 25 min. The loss of nine volatile esters and a decrease in the total flavour were found in canned jackfruit puree as compared to fresh jackfruit puree.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENCIRIAN PERISA LIMA KULTIVAR BUAH NANGKA (*ARTOCARPUS HETEROPHYLLUS* L.) DAN PENGOPTIMUMAN PROSES PENGETINAN PURI BUAH NANGKA

Oleh

ONG BEE TEIN

Mei 2006

Pengerusi: Nazimah Sheikh Abdul Hamid, PhD

Fakulti: Sains dan Teknologi Makanan

Kajian ini adalah mengenai pencirian perisa lima kultivar buah nangka (*Artocarpus heterophyllus* L.) dan mengoptimumkan proses penghasilan puri buah nangka yang ditinkan. Di dalam bahagian pertama kajian ini, dua puluh tiga sebatian meruap diekstrak menggunakan cara pengekstrakan pelarut diklorometana dan dikenalpasti secara tentatif dengan kromatografi gas (GC) dan kromatografi gas-spektrometri jisim (GC-MS). Pembentukan sebatian meruap meningkat semasa proses peranakan. Penghasilan sebatian meruap yang baru dalam jumlah yang sedikit pada hari ketiga selepas dituai menandakan proses peranakan buah nangka telah bermula. Data yang didapati menunjukkan bahawa peranakan buah nangka adalah optimum pada hari kelima selepas dituai. Semasa peranakan, variasi sebatian meruap di antara bahagian buah (atas, tengah dan bawah) yang berbeza adalah terlalu sedikit untuk memberikan nilai yang signifikan.

Profil sebatian meruap dalam lima kultivar buah nangka telah dikenalpasti menggunakan ruang tutupan (*headspace*) pengekstrakan mikro fasa pepejal (*Solid Phase Microextraction*) dan kromatografi gas-spektrometri jisim masa penerbangan (*Gas Chromatography-Time Of Flight Mass Spectrometry*). Analisis kualitatif dan kuantitatif telah dijalankan dengan menggunakan gentian divinilbenzene/carboxen/polidimetilsiloxane (DVB/CAR/PDMS) dengan masa pengekstrakan selama 10 minit. Tiga puluh tujuh sebatian telah dikenalpasti daripada lima kultivar nangka yang dikaji. Aroma pencirian yang mempunyai kepekatan yang tinggi dan menyumbang kepada perisa nangka adalah etil isovalerat, 3-metilbutil asetat, 1-butanol, propil isovalerat, isobutil isovalerat, 2-metilbutanol dan butil isovalerat. Kemunculan sebatian ini dalam kesemua kultivar buah nangka menyumbang kepada perisa manis dan buah dalam buah nangka. Kepekatan sebatian meruap ini memainkan peranan yang penting dalam penentuan perisa keseluruhan untuk setiap kultivar. Setiap kultivar turut mempamerkan sebatian yang unik yang dapat membezakan satu kultivar daripada yang lain.

Semasa pengetinan puri nangka, pengoptimuman masa (10 – 30 minit) dan suhu (100 – 120°C) telah dikaji dengan menggunakan kaedah respon permukaan (*response surface methodology*). Nilai respon didapati sesuai untuk model polinomial arahan kedua. Kesan pembolehubah bebas ke atas kemanisan, pH dan jumlah pepejal larut adalah tidak signifikan ($p > 0.05$). Analisis varians (ANOVA) dan pekali regresi menunjukkan kesan kadar langsung positif yang signifikan akibat kesan suhu terhadap perisa masak

($p < 0.01$), kemasaman ($p < 0.05$), kepahitan ($p < 0.05$) dan warna ($p < 0.05$), manakala perisa buah ($p < 0.05$), kepekatan ($p < 0.01$) dan kroma ($p < 0.001$) menunjukkan kesan kadar langsung negatif. Masa pemprosesan hanya mempengaruhi perisa masak ($p < 0.05$) dan warna ($p < 0.05$) puri nangka. Model optimum ini menghasilkan julat yang diingini untuk pemprosesan yang optimum, iaitu julat suhu di antara 100 hingga 110°C sementara julat masa pemprosesan adalah di antara 15 hingga 25 minit. Kehilangan sembilan ester meruap dan penurunan kandungan perisa didapati dalam puri nangka yang ditinkan berbanding dengan puri nangka yang segar.

ACKNOWLEDGEMENTS

First and foremost, I would like to express my deepest appreciation and gratitude to my supervisor, Dr. Nazimah Sheikh Abdul Hamid, and my co-supervisors, Associate Professor Dr. Azizah Osman, who has not only given me invaluable advice and guidance from their vast experience, but also for their precious time and supervision. I would also like to extend my heartfelt thanks to Encik Dzulklifi Mat Hashim, for his uncountable advice, time and constructive suggestions throughout the course of my research study.

I'm forever indebted to my mother and father, for their infinite support, love, care and understanding. I am also very thankful for having brothers who were ever so helpful and caring during the difficult times. I would also like to extend my greatest appreciation to my boyfriend, Keng Fei who had been with me through the hard times with his patience and support throughout all these years of studies.

I'm also indebted to Sung Tong, Yit Yang, Chun Kiat, Tuck Keong and Horng Eng for their infinite support, help and their friendship; without them, working in the lab would not have been as enjoyable and memorable.

I would also like to express my deepest appreciation and thanks to all the staff of Department of Food Science and Technology for providing the research facilities and technical assistance. I would like to acknowledge the financial support provided by IRPA grant (01-02-04-0789 EA001) awarded to Dr. Nazimah Sheikh Abdul Hamid for

this study. Acknowledgement is also due to the GRA scholarship for granting me the opportunity to pursue my master degree.

To all my fellow friends, your companion and care has helped me pulled through those difficult times. Remember that you will always have a special place in my heart.

I certify that an Examination Committee has met on 31 May 2006 to conduct the final examination of Ong Bee Tein on her Master of Science thesis entitled "Flavour Characterization of Jackfruit (*Artocarpus heterophyllus* L.) from Five Cultivars and Optimization of Canning Conditions for Jackfruit Puree" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

Russly Abdul Rahman, PhD

Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

Jinap Selamat, PhD


Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

Tan Chin Ping, PhD

Lecturer
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

Osman Hassan, PhD

Associate Professor
School of Chemical Science and Food Technology
Universiti Kebangsaan Malaysia
(External Examiner)


HASANAH MOHD. GHAZALI, PhD
Professor/Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 28 AUG 2006

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

Nazimah Sheikh Abdul Hamid, PhD

Lecturer

Faculty of Food Science and Technology

Universiti Putra Malaysia

(Chairman)

Azizah Osman, PhD

Associate Professor

Faculty of Food Science and Technology

Universiti Putra Malaysia

(Member)



AINI IDERIS, PhD

Professor/Dean

School of Graduate Studies

Universiti Putra Malaysia

Date: **14 SEP 2006**

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.


ONG BEE TEIN

Date: 20 AUG 2006

TABLE OF CONTENTS

	Page
DEDICATION	2
ABSTRACT	3
ABSTRAK	6
ACKNOWLEDGEMENTS	9
APPROVAL	11
DECLARATION	13
LIST OF TABLES	17
LIST OF FIGURES	19
LIST OF ABBREVIATIONS	21
 CHAPTER	
 I GENERAL INTRODUCTION	 24
 II LITERATURE REVIEW	 28
Jackfruit (<i>Artocarpus heterophyllus</i> L.)	28
Characteristics of the fruit and tree	29
Harvest maturity	30
Chemical composition of jackfruit	32
Flavour volatiles and its importance in tropical fruits	33
Factors that Affects Flavour of Fruits and Vegetables	38
Volatiles in jackfruit	50
Tropical fruit puree and its application	54
Thermal Processing	56
Canning as a method of preservation for fruit puree	56
Effect of thermal processing on the properties of fruit products	58
Rheological properties of fruit puree	61
Flavour extraction	67
Liquid-liquid / solvent extraction	74
Solid Phase Microextraction (SPME) method	75
Measurements of Flavour Volatiles	80
Quantitative description analysis (QDA)	81
Response Surface Methodology (RSM)	82
 III FLAVOUR CHANGES DURING RIPENING OF JACKFRUIT	 84
(<i>Artocarpus heterophyllus</i> L.) CULTIVAR J3	
Introduction	84
Materials and Methods	86
Plant materials	86
Sampling of fruit	87
Flavour extraction	87
Gas Chromatography analysis	88

Gas Chromatography-Mass Spectrometry analysis	88
Statistical analysis	89
Results and Discussions	90
Conclusions	96
IV ANALYSIS OF VOLATILE COMPOUNDS IN FIVE JACKFRUIT (<i>Artocarpus heterophyllus</i> L.) CULTIVARS USING SOLID PHASE MICROEXTRACTION (SPME) AND GAS CHROMATOGRAPHY – TIME OF FLIGHT MASS SPECTROMETRY (GC-TOFMS)	98
Introduction	98
Materials and methods	100
Materials	100
Plant materials	101
Standards and sample preparations	101
Solid phase microextraction sampling	102
Separation and detection – SPME/GC-TOFMS	102
Statistical analysis	103
Results and discussions	104
Selection of fiber and extraction time	104
Volatile compounds of jackfruit cultivars	107
Conclusions	116
V OPTIMIZATION OF CANNING CONDITIONS FOR THE PRODUCTION OF JACKFRUIT (<i>Artocarpus heterophyllus</i> L.) PUREE USING RESPONSE SURFACE METHODOLOGY	117
Introduction	117
Materials and methods	120
Materials	120
Puree preparation	121
Sterilization processes	121
Quantitative descriptive analysis	122
Viscosity determination	123
Flavour volatiles analysis	124
Colour, pH and total soluble solids determinations	126
Experimental design and statistical analysis	127
Results and discussions	130
Fitting the model	130
Effect of different canning conditions on sensory evaluation	134
Effect of different canning conditions on physical properties	139
Optimization	143
Conclusions	145
VI GENERAL CONCLUSIONS AND RECOMMENDATIONS	146
Conclusions	146
Recommendations	148

REFERENCES/BIBLIOGRAPHY	151
APPENDICES	179
BIODATA OF THE AUTHOR	199

LIST OF TABLES

Table		Page
1	List of volatile compounds identified in jackfruit (<i>Artocarpus heterophyllus</i> L.) by Sword et al. (1978), Rasmussen (1983) and Maia et al. (2004)	53
2	Processing parameters of various fruit and vegetable products in literature	59
3	Published data on extraction and analysis methods used in determination of volatile flavour compounds of various fruits and its products	68
4	Volatile compounds of <i>A. heterophyllus</i> , cultivar J3 tentatively identified using Supelcowax-10 capillary column	90
5	Coefficient of variation (CV) on the peak areas of selected volatile compounds detected in the three fibers after triplicate extraction	106
6	Concentrations of flavour volatile compounds detected in cultivars J31, J1, J6, J30 and J3 using DVB/CAR/PDMS fiber	109
7	Volatile compounds and their flavour descriptors of five jackfruit (<i>Artocarpus heterophyllus</i> L.) cultivars separated based on principal components 1, 2 and 3	112
8	Design of experimental runs and experimental data for the dependent variables sensory evaluation, viscosity, colour, total soluble solids and pH of jackfruit puree under different canning conditions	129
9	Analysis of variance on independent variables as linear, quadratic and interaction terms on the response variables	131
10	Regression coefficients of predicted quadratic polynomial models for the ten responses of jackfruit puree	133
11	Volatile flavour compounds identified in fresh and processed jackfruit puree	136
12	Percent loss in concentration of important volatile ester compounds in jackfruit puree under different canning conditions	137

13	Consistency coefficient, K , flow behaviour, η and r^2 in Power-Law equation at different processing time and temperature combination	142
14	Evaluation of microbial growth and spore formers in canned jackfruit puree under a series of canning condition after storing at accelerated temperature (40 °C) for six months	144
A-1	Numerical values for scores plot of PC1 and PC2 in the analysis of flavour changes during jackfruit ripening	180
A-2	Numerical values for loadings plot of PC1 and PC2 in the analysis of flavour changes during jackfruit ripening	180
A-3	Numerical values for loadings plot of PC1, PC2 and PC3 in the analysis of flavour in five different cultivars of jackfruit	181
A-4	Numerical values for scores plot of PC1, PC2 and PC3 in the analysis of flavour in five different cultivars of jackfruit	182

LIST OF FIGURES

Figure		Page
1	A general schematic of the biosynthetic pathways for volatile aromas of plant products	42
2	Chromatograms of flavour compounds from middle portions of jackfruit cultivar J3 at days 1, 3, 5, and 6 after harvest	92
3	The score plots of changes in volatile compounds during ripening process	93
4	The X-loadings of volatile compounds present in fruits from days 1, 3, 5 and 6 after harvest	94
5	Comparison of different SPME fibers (PDMS, PDMS/DVB and DVB/CAR/PDMS) after extraction for 10 min at 30°C and separated using Supelco Wax-10 (10 m x 0.1 mm i.d., 0.1µm film thickness)	105
6	Effect of extraction time at 30°C on the extraction efficiency of flavour compounds using DVB/CAR/PDMS fiber	107
7	Bi-plots of principal components 1 and 2 of the volatile compounds of five jackfruit cultivars	111
8	Bi-plots of principal components 1 and 3 of the volatile compounds of five jackfruit cultivars	114
9	Response surface plot for the effect of independent variables on the sensory quality (a) fruity flavour, (b) cooked flavour, (c) sourness and (d) bitterness of processed jackfruit puree as significantly affected (at $p < 0.05$) by the canning conditions	135
10	Response surface plot of (a) hue, (b) chroma and (c) viscosity of the processed jackfruit puree as significantly affected (at $p < 0.05$) by the canning conditions	140
11	Behaviour of processed jackfruit puree as a solid	141
12	Superimposed contour plots for optimization of significant response variables of fruity flavour, cooked flavour, sourness, bitterness, hue,	144

chroma and viscosity

B-1	Sensory evaluation forms for discrimination test during screening of respondents. Similar forms were generated for sweetness, saltiness, bitterness and astringency	183
B-2	Odour description test form for screening of respondents	184
B-3	Texture description test form for screening of panelist	185
B-4	Terms of attributes provided to panelist during round table training sessions	186
B-5	Sensory evaluation forms for panelist during sample assessments	188

LIST OF ABBREVIATIONS

%	percent
°C	degree centigrade
°F	degree Fahrenheit
μl	microliter
μm	micrometer
a*	colorimetric a*
ANOVA	analysis of variance
b*	colorimetric b*
C	carbon chain
CI	chemical ionization
cm	centimeter
CV	coefficient of variation
FDHS	Dynamic Headspace Sampling
DVB/CAR/PDMS	divinylbenzene/carboxen/polydimethylsiloxane
EI	electron ionization
eV	electron voltage
FC	Flash Chromatography
g	gravity
G'	storage modulus
G''	loss modulus
GC	Gas Chromatography

GC/FTIR	Gas Chromatography/Fourier transform infrared
GC/IR	Gas Chromatography / Infrared
GC/MS	Gas Chromatography / Mass Spectrometry
GC/O	Gas Chromatography / Olfactometry
GC-FID	Gas Chromatography – Flame Ionization Detector
GC-TOFMS	Gas Chromatography – Time of Flight Mass Spectrometry
GLC	Gas Liquid Chromatography
GLC/MS	Gas Liquid Chromatography / Mass Spectrometry
HRGC	Capillary Gas Chromatography
HRGC/MS	Capillary Gas Chromatography / Mass Spectrometry
hrs	hours
i.d.	internal diameter
kg	kilogram
L	liter
L*	lightness
LLE	Liquid-Liquid Extraction
m	meter
<i>m/z</i>	mass spectra
min	minute
min-l	per minute
ml	milliliter
mm	millimeter
mPa	mili Pascal

ND	not detected
PC	principal component
PDMS / DVB	polydimethylsiloxane / divinylbenzene
PDMS	polydimethylsiloxane
ppm	parts per million
PTR-MS	proton transfer reaction – mass spectrometry
QDA	quantitative descriptive analysis
rad/s	radius per second
RH	relative humidity
rpm	revolution pre minute
RSM	response surface methodology
s	second
s ⁻¹	per second
SDE	Simultaneously Distillation Extraction
SDEV	Simultaneously Distillation-Extraction under Pressure
SPME	Solid Phase Microextraction
TLC	Thin Layer Chromatography
VHS	Vacuum Headspace Sampling

CHAPTER I

GENERAL INTRODUCTION

Jackfruit, a dicotyledonous compound fruit of the jacktree (*Artocarpus heterophyllus* L.), is grown in most tropical countries such as Sri Lanka, Burma, Malaysia, Indonesia and Brazil, but it is particularly abundant in India and Bangladesh. The complex fruit, which matures 4 to 5 months, turn yellowish green with a golden yellow flesh surrounded by yellow fibers. Jackfruit is consumed both as a vegetable in the unripe stage and also as a fruit when ripe. The popularity of jackfruit as a commercial fruit has been restricted to the growing regions. Generally, there is very little research available on jackfruit. The gross composition of jackfruit, its vitamin content (Bose, 1985; Ahmed et al., 1986; Bhattacharjee, 1986), water-soluble sugar (Wills et al., 1986; Selvaraj and Pal, 1989), starch (Bobbio et al., 1978; Hossain et al., 1990; Rahman et al., 1995; Rahman et al., 1999), free sugar and fatty acids (Chowdhury et al., 1997), and flavour volatiles (Swords et al., 1978; Rasmussen, 1983; Maia et al., 2004) have been documented.

Maturity indices for various horticultural crops have relied on different features of the commodity, such as duration of development, size, colour, firmness, etc., which provide an adequate estimation of maturity (Shewfelt, 1993). Mature jackfruit contains between 35-40% edible flesh (Crane, 2002). However, it is not easy to determine the exact time when the fruit is ripe. Most fruits do not have characteristic aromas or flavours